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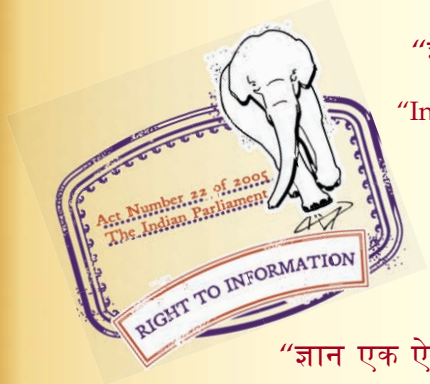
IS 10512 (2003): Method for Determination of Wax Content in Bitumen [PCD 6: Bitumen Tar and their Products]

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भारतीय मानक

# बिटुमन में मोम का अंश ज्ञात करने की पद्धति—विशिष्टि

( पहला पुनरीक्षण )

*Indian Standard*

# METHOD FOR DETERMINATION OF WAX CONTENT IN BITUMEN—SPECIFICATION

*( First Revision )*

ICS 75.140

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BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

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Price Group 4

FOREWORD

This Indian Standard (First Revision) was adopted by the Bureau of Indian Standards, after the draft finalized by the Bitumen Tar and Their Products Sectional Committee had been approved by the Petroleum, Coal and Related Products Division Council.

This standard was published in 1983, which was largely based on UOP 46-64. Since the present method is having the following disadvantages the Committee decided to revise the standard to align the same with DIN 52015:1980 'Determination of paraffin wax content in bitumen' in order to over come the disadvantages:

a) The method is not specific to 'Paraffin wax content' only but specifies the wax content which includes wax and micro-crystalline wax,

b) The method does not mention the purity of 'fullers earth',

c) There is no mention of 'Reproducibility' in the method,

d) Fullers earth having the particle size within the range 10-35 μ, is not available indigenously,

e) There is no mention about the availability of fullers earth,

f) The sieves required to check the particle size of fullers earth are not available indigenously, and

g) The wax content value varies with the quality of fullers earth.

The composition of the Committee responsible for formulation of this standard is given in Annex A.

In reporting the results of a test or analysis made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'.

# *Indian Standard*

# METHOD FOR DETERMINATION OF WAX CONTENT IN BITUMEN—SPECIFICATION

## *( First Revision )*

### 1 SCOPE

This standard prescribes a method used for determining the paraffin wax content of bitumen. The method is applicable to bitumens having paraffin wax with melting point above 25°C.

NOTE—Paraffin waxes include only those hydrocarbons crystallizing in an ether/ethanol mixture at temperatures down to –20°C, obtained by a specified process and having a melting point of above 25°C.

### 2 REFERENCE

The following standard contains provisions which through reference in this text, constitutes provisions of this standard. At the time of publication the edition indicated was valid. All standards are subject to revisions and parties to agreements based on this standard are encouraged to investigate the possibility of applying the most recent edition of the standard indicated below:

| *IS No.* | *Title* |
|---|---|
| 334 : 2002 | Glossary of terms relating to bitumen and tar (*third revision*) |

### 3 TERMINOLOGY

For the purpose of this standard, the definitions given in IS 334 shall apply.

### 4 APPARATUS

**4.1 Balance**, readable and accurate to ± 0.5 mg.

**4.2 Steam Bath**

**4.3 Heating Cabinet (Oven)**, for temperature at least 150°C.

**4.4 Bunsen Burner**

**4.5 Thermometers**, having least count at least 0.5°C.

**4.6 Distillation Flask**, as shown in Fig. 1, fitted with cork stopper.

**4.7 Sheet Metal Guard Ring**, of about 18 mm inside diameter and about 65 mm outside diameter.

**4.8 Cooling Bath**, as shown in Fig. 2.

**4.9 Test Tubes**, as shown in Fig. 2, fitted with spout and bored cork stopper.

**4.10 Funnel**, as shown in Fig. 2.

**4.11 Erlenmeyer Flask**, 100 ml capacity, fitted with a bored cork stopper (to be used as distillation receiver) (*see* Fig. 3).

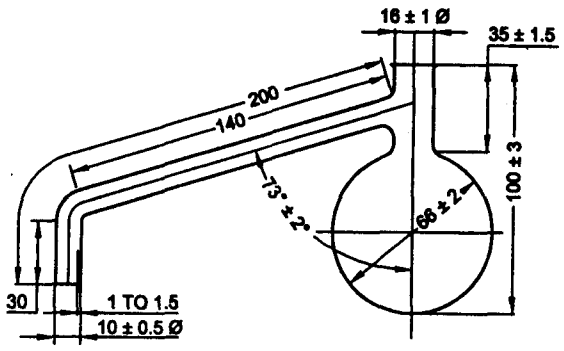
**4.12 Test Tube**, fitted with ground socket and a wash bottle head fitted with ground cone (*see* Fig. 4).

**4.13 Filtering Flask**, 500 ml capacity.

**4.14 Vacuum Pump**

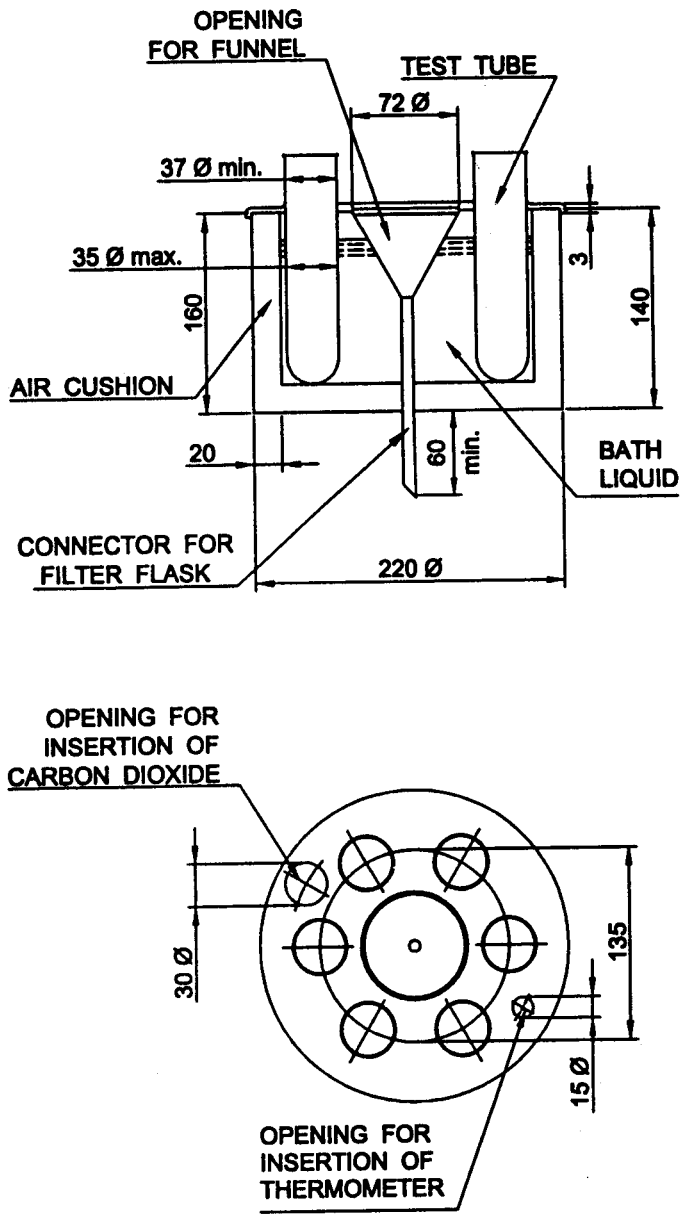
**4.15 Evaporating Dish**

NOTE—The apparatus for determining the paraffin wax content is available with M/s Petrotest Instruments GmbH & Co KG, Ludwig-Erhard-Ring 13, D-15827, Dahlewitz.




All dimensions in millimetres.

FIG. 1 DISTILLATION FLASK




All dimensions in millimetres.

FIG. 2 COOLING BATH

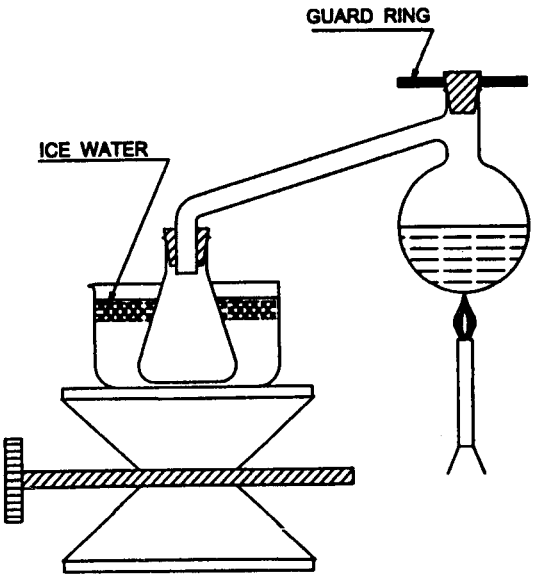



FIG. 3 SCHEMATIC DIAGRAM OF DISTILLATION ARRANGEMENT

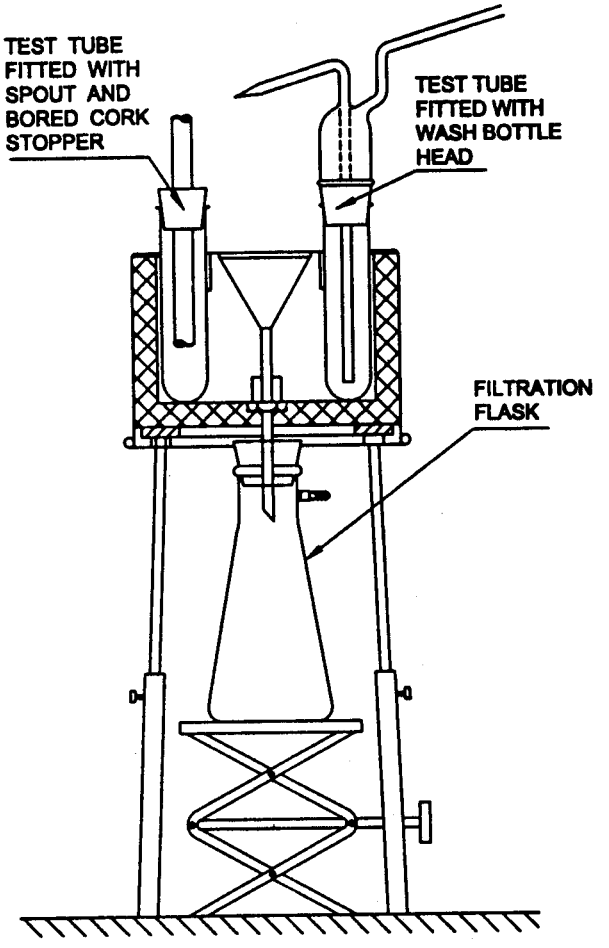



FIG. 4 SCHEMATIC DIAGRAM OF FILTRATION ARRANGEMENT

## 5 REAGENTS

**5.1 Anhydrous Diethyl Ether,** referred to in this standard as ether.

**5.2 Ethanol, Absolute**

**5.3 Reagent Grade Acetone**

5.4 Petroleum spirit having the following characteristics:

| | |
|---|---|
| a) Density at 15 °C, g/ml | : 0.690-0.705 |
| b) Boiling range: | |
| 1) Initial boiling point, °C , *Min* | : 65 |
| 2) Collected volume of distillate, about upto 90 °C, percent by volume | : 90 |
| 3) Drying point, °C, *Max* | : 95 |
| c) Aniline point, °C | : 59 to 61 |

## 6 PROCEDURE

**6.1** Melt the bitumen sample, pour 25 ± 1g into the distillation flask and weigh to the nearest 10 mg ( $M_A$). Heat the distillation flask with a 150 mm high flame, that has just ceased to be luminous, in such a way that the first distillate drop is produced within 3 to 5 min. The guard ring, loosely fitted on the distillation flask, shall prevent possible burning of the cork stopper. Take care that the vapours produced during distillation are largely condensed. For this purpose, the distillation receiver, weighed to the nearest 10 mg, into which the lower bent end of the outlet tube projects to its full length (*see* Fig. 3), shall be immersed as far as possible into a mixture of finely reduced ice and water. There shall, however, still be a field of vision to allow the rate of distillation to be checked. Adjust this rate so that a drop falls from the end of the outlet tube into the distillation receiver approximately every second. When no more drops are produced over a period of 10 s, continue heating for a further 1 min with a completely non-luminous flame until the flask glows red. Complete the distillation in a maximum of 15 min. Do not transfer the condensate left in the outlet tube after distillation to the distillation receiver. Mix the distillate thoroughly by gently warming it whilst at the same time carefully swirling the receiver.

**6.2** After cooling, weigh the distillate contained in the receiver to the nearest 10 mg ( $M_B$). Depending on the expected paraffin wax content, weigh to the nearest 2 to 4 g ± 5 mg (mass $M_C$) of the warm distillate into the test tube (provided with spout). If the paraffin wax content cannot be estimated in advance, an initial mass of approximately 3 g is recommended.

**6.3** Dissolve the initial mass of distillate in 25 ± 1 ml of ether and add 25 ± 1 ml of ethanol. Close the test tube with a stopper fitted with a thermometer extending down into the liquid and place the test tube in the cooling bath. Cool the bath liquid by adding finely reduced solid carbon dioxide to get sample temperature of −20 ± 0.5°C. A low temperature bath may be used. Transfer 20 ± 1 ml of an ether/ethanol mixture (as the washing liquid), prepared in a 1:1 ratio, in the test tube fitted with the wash bottle head and cool it in the cooling bath to −20 ± 0.5 °C.

**6.4** Keep the sample temperature −20 ± 0.5 °C constant until filtration is complete. Place the round filter paper in the funnel standing in the cooling bath and connect it to the filter flask. Quickly transfer the slurry of crystals produced at −20 ± 0.5°C to the filter. Rinse the test tube with the cooled washing liquid. Re-adjust the temperature of the washing liquid to −20 ± 0.5 °C and use it again for rinsing the crystal slurry in the filter. Distribute the washing liquid as uniformly as possible between the three washing operations.

**6.5** Support the filtration by a gentle suction process during which the vacuum pressure shall not fall below 5 kPa (50 mbar). As soon as no more filtrate is dropping through, disconnect the filter assembly with vacuum pump, lift off the filter using the pincers and place it in the funnel situated over the evaporating dish, which has been previously weighed to the nearest 0.5 mg. Dissolve the crude paraffin residue by carefully spraying heated petroleum spirit over it. Dissolve in the same way any paraffin that may be adhering to the thermometer or to the test tube. Evaporate the mixed filtrates in the evaporating dish over the steam bath. To prevent the liquid creeping over the rim, carry out the evaporation in a weak air or nitrogen stream. Dry the residue for 15 ± 1 min at 125 ± 5 °C in the heating cabinet and then allow it to cool. As soon as the previously purified paraffin waxes have cooled down but have not quite solidified, add 15 ml of acetone.

**6.6** Dissolve the paraffin waxes by gently heating and carefully swirling the evaporation dish. Make up any acetone lost by evaporation. Cool the acetone/paraffin wax solution in a water bath to 15 ± 0.5 °C and separate by filtering the paraffin waxes. Then wash the evaporating dish, the thermometer and the filter several times with acetone brought to 15 ± 0.5 °C from a wash bottle. The total volume of washing liquid shall be 30 ± 1 ml. Dissolve the paraffin waxes purified in this way, by carefully spraying them with heated petroleum spirit and collect them again in the evaporating dish already used. Then evaporate the collected liquid in a weak air flow or nitrogen stream over the steam bath.

**6.7** Dry the crystallized paraffin waxes obtained, at 125 ± 5 °C for 15 ± 1 min in the heating cabinet and after cooling in the desiccator weigh them to the nearest 0.5 mg ( $M_D$). This final mass shall be between 50 and 100 mg. Otherwise, reject this result and repeat the test with an appropriately changed initial mass of the same distillate ($M_C$).

**6.8** Determine the solidification point of paraffin wax on the rotating thermometer.

## 7 CALCULATION

**7.1** For each test portion, calculate the paraffin wax content, expressed as a percentage by mass, using the following equation:

$$\text{Paraffin wax, percent } M = \frac{(M_B \times M_D)}{(M_A \times M_C)} \times 100$$

where

$M_A$ = initial mass of bitumen, in g;
$M_B$ = mass of distillate received, in g;
$M_C$ = initial mass of distillate taken up for test, in g; and
$M_D$ = final mass of paraffin wax, in g.

**7.2** If the values measured for both test portions do not differ by more than 0.3 percent by mass, determine the mean of the two values. Otherwise, carry out a determination on a third test portion of 25 g and take the mean of the two values being the closest together. These values, however, shall not differ by more than 0.3 percent by mass. If the first two values are equidistant from the third, specify the third value.

**7.3** If it is not possible to obtain a mean value from these three values under the specified conditions, reject all the three values and repeat the test on two further test portions.

**7.4** Express the paraffin wax content as a percentage by mass, rounded to the nearest 0.1.

## 8 PRECAUTION

Never evaporate petroleum spirit on naked flame or on a hot plate. Steam bath should invariably be used.

## 9 PRECISION

### 9.1 Repeatability

If two results are obtained by one and the same operator under repeatability conditions, they are considered to be acceptable and in accordance with the specifications of this standard if they do not differ by more than 0.3 percent by mass.

### 9.2 Reproducibility

If two single and independent results are obtained in two different laboratories under comparable conditions, they are considered to be acceptable and in accordance with the specifications of this standard if they do not differ by more than 1.0 percent by mass.

# ANNEX A

(*Foreword*)

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